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REVIEW

Corrosion inhibition of carbon steel immersed in a 1 M HCl solution using benzothiazole derivatives



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KEYWORDS

Steel ST-37; Immersion time; EIS; Corrosion inhibitor; AFM **Abstract** Corrosion inhibition effect of 2-mercaptobenzothiazole (MBT) and 2-aminobenzothiazole (ABT) compounds on ST-37 carbon steel in 1 M hydrochloric acid solution was investigated by electrochemical impedance spectroscopy (EIS), and it was observed that both of these compounds have corrosion inhibition effect on carbon steel. Evaluation of electrochemical behavior in test solutions showed that by increasing the immersion time from 15 to 300 min, corrosion resistance of samples is increased and at the same immersion time MBT has a better corrosion inhibition in comparison to ABT. AFM technique was performed for MBT and ABT. The results of calculations showed superior inhibition efficiency of MBT in comparison to ABT. This can cause easier protonation and consequently adsorption on the metal surface occurs.

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1. Introduction

Carbon steel is a common constructional material for many industrial units because of its low cost and excellent mechanical properties. However, it suffers severe attack in Acidic solutions which are used extensively in chemical and several industrial processes such as acid pickling, acid cleaning, acid descaling, etc.

Chemical inhibitors are often used for these processes mainly to control the metal dissolution and acid consumption, the selection and application of inhibitors are actually complicated because of the variable corrosive environments in these processes (Alsabagh et al., 2006; Prabhu et al., 2008). For proper selection of inhibitors, mechanistic information on corrosion and inhibition processes is needed. The search for new and efficient corrosion inhibitors requires the clarification of interactions between inhibitor molecules and metal surfaces (Atta et al., 2011).

The use of organic molecules as corrosion inhibitor is one of the most practical methods for protecting metals against the corrosion and it is becoming increasingly popular. The existing data show that organic inhibitors act by the adsorption and protect the metal by film formation. Organic compounds bearing heteroatoms with high electron density such as phosphor, sulfur, nitrogen, oxygen or those containing multiple bonds which are considered as adsorption centers, are effective as corrosion inhibitor (Döner et al., 2011). Among the inhibitors which are effective in acid solutions there are nitrogen containing compounds (such as derivatives of amines pyridazine, quinoline and pyridine, pyrazole, pyrazine, acridine, benzimidazole and triazole) and mercapto functional compounds (Most of the mercapto functional azole compounds have shown mixed-type corrosion inhibition in acidic solutions for protection of mild steel) (Quartarone et al., 2012; Mahdavian and Ashhari, 2010). In literature review benzothiazoles have been used as corrosion inhibitors for steel (Quraishi et al., 1996; Al-Mayouf et al., 1998; Fouda et al., 2013; Parameswari et al., 2012) and copper (Zhou et al., 1993; Nandeesh and Sheshadri, 1988; Vastag et al., 2001), but the studies on corrosion of zinc are sparse.

A detailed understanding of the mechanism of adsorption for corrosion inhibitors requires the characterization of corrosion inhibitor films by using surface analytical techniques. Electrochemical Impedance Spectroscopy (EIS) has proved to be a powerful technique to study corrosion processes and inhibitor performance in different environments (Desimone et al., 2011).

The aim of this work is to study the effect of 2-mer-captobenzothiazole (MBT) and 2-aminobenzothiazole (ABT) on inhibition properties on the corrosion of ST-37 Carbon steel in 1 M HCl by using techniques like AFM and electrochemical impedance calculations during immersion time.

2. Experimental

2.1. Materials

The ST-37 low carbon steel used had the following chemical composition (wt%): 0.11C, 0.56Mn, 0.03Si, 0.007P, 0.005S, 0.07Cr, 0.03Ni and balance Fe (Jafarzadegan et al., 2012).

Benzothiazoles (BTs), a group of xenobiotic compounds containing a thiazole (Th) ring fused with benzene ring, have a wide range of biological properties (Sayyah and Azooz, 2016). BTs are applied as fungicides, herbicides, antialgal agents, slimicides, rubber additives, corrosion inhibitors and chemotherapeutics.

2-mercaptobenzothiazole (MBT) (Fig. 1) is known to form metal complexes with various metal ions, including Fe, Cu, Cd, Bi, Pb, Ag, Hg, Pd, Pt, Zn and Co. Due to their unique chemical reactivity with different metal and mineral surfaces, MBT and their derivatives have been used widely in many industrial and engineering applications, including corrosion inhibitors for copper, iron or their alloys. For example, MBT has been used as collectors for flotation of copper sulfide, lead sulfide, activated zinc sulfide, nickel sulfide and gold-bearing pyrite (Liu et al., 2012).

2-Aminobenzothiazole (ABT) (Fig. 1) is applied for its local anesthetic action in human and veterinary medicine and it is also used as a reactant in the production of azo dyes (Lewandowski et al., 2011).

2.2. Methods

The apparatus for electrochemical investigations consists of computer controlled Auto Lab potentiostat/galvanostat (PGSTAT302N). The electrochemical experiments were carried out using a conventional three electrode cell assembly at 25 ± 2 °C. A rectangular platinum foil was used as counter electrode and saturated calomel electrode as the reference electrode. The EIS experiments were conducted in the frequency range of 100 kHz to 0.01 Hz at open circuit potential. The AC potential amplitude was 10 mV.

Two ST-37 low carbon steel were mounted with an exposed surface area of 1 cm². Before each electrochemical experiment, surfaces were polished with 220, 400, 600, 800, 1000, and 1200 grades of emery paper, degreased with acetone and rinsed by distilled water. ST-37 samples were immersed in acidic solution containing 2×10^{-3} M inhibitor. Time interval of 20–25 min

Figure 1 Molecular structures of the investigated compounds.

was given for steady state attainment of open circuit potential. Then the electrochemical impedance spectroscopy (EIS) tests were performed on them at different times (15, 75,120, 240 and 300 min). MBT and ABT were purchased from Merck and used as inhibitor. Fitting of experimental impedance spectroscopy data to the proposed equivalent circuit was done by means of home written least square software based on the Marquardt method for the optimization of functions and Macdonald weighting for the real and imaginary parts of the impedance (Aghassi et al., 2011; Quartarone et al., 2012).

For surface analysis, the specimens were immersed in 1 M HCl prepared with and without addition of 2×10^{-3} M of inhibitors at 25 ± 2 °C for 5 h, cleaned with distilled water. The surface morphology of the electrode surface was evaluated by atomic force microscopy (AFM) Nan Surfeasyscan2.

3. Results and discussion

3.1. Electrochemical impedance spectroscopy (EIS)

In order to get more information about the corrosion phenomena, solution resistance (R_s) , charge transfer resistance (R_{ct}) , double layer capacitance (C_{dl}) of steel electrode, impedance measurements have been carried out in 1 M HCl solutions in the absence and presence of MBT and ABT at different immersion times at the open circuit potential (OCP). The Nyquist plot for steel sample in 1 M HCl solution with 2×10^{-3} M MBT and ABT at the different immersion times measured at open circuit potential is shown in Figs. 2 and 3a. The data reveal that impedance diagram consists of a depressed capacitive loop due to charge transfer resistance and double layer capacitance

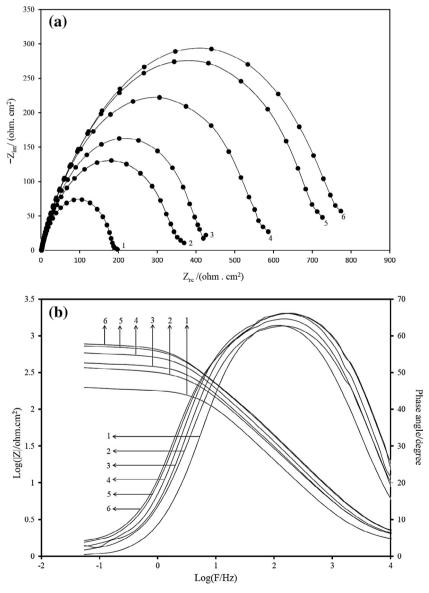


Figure 2 EIS spectra of carbon steel for the blank test and inhibition test at the concentration of 2×10^{-3} in 1 M HCl for ABT (a) Nyquist plot; (b) Bode plots: (1) blank, (2) 15 min, (3) 75 min, (4) 120 min, (5) 240 min and (6) 300 min.

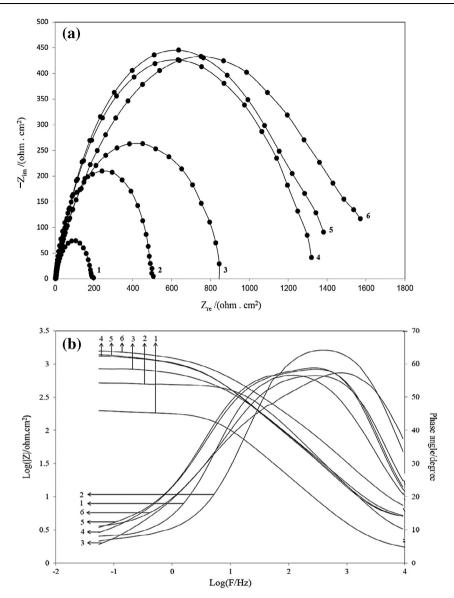


Figure 3 EIS spectra of carbon steel for the blank test and inhibition test at the concentration of 2×10^{-3} in 1 M HCl for MBT (a) Nyquist plot; (b) Bode plots: (1) blank, (2) 15 min, (3) 75 min, (4) 120 min, (5) 240 min and (6) 300 min.

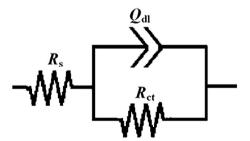


Figure 4 Equivalent circuits compatible with the experimental impedance data in Figs. 2 and 3 for corrosion of steel electrode in 1 M hydrochloric acid solution.

which indicates that the corrosion of steel is mainly controlled by a charge transfer process. Bode phase plots are shown in Fig. 2b and Fig. 3b where one well resolved peak is observed due to charge transfer process pointing to depressed semicircle in Nyquist plot. The equivalent circuit compatible with the Nyquist diagram recorded in the presence of alcohols is depicted in Fig. 4. The simplest approach requires the theoretical transfer function $Z(\omega)$ to be represented by a parallel combination of a resistance R_{ct} and a capacitance C, both in series with another resistance R_s (Danaee et al., 2008):

$$Z(\omega) = R_s + \frac{1}{\frac{1}{R_s} + i\omega C} \tag{1}$$

 ω is the frequency in rad/s, $\omega = 2\pi f$ and f is frequency in Hz. To obtain a satisfactory impedance simulation of steel, it is necessary to replace the capacitor (C) with a constant phase element (CPE) Q in the equivalent circuit. The most widely accepted explanation for the presence of CPE behavior and depressed semicircles on solid electrodes is microscopic roughness, causing an inhomogeneous distribution in the solution resistance as well as in the double layer capacitance (Danaee

Table 1 Electrochemical impedance parameters for ST-37 carbon steel in aerated 1 M HCl solution with 2×10^{-3} M of MBT after different immersion times.

Immersion time	$R_s (\Omega \text{ cm}^2)$	$R_{ct} (\Omega \text{ cm}^2)$	$Q_{dl} \times 10^{-6} (F cm^{-2})$	n	η%	$C_{dl} \times 10^{-6} \; (\text{F cm}^{-2})$
Blank	1.528	193.2	253	0.8143	0	126.9861
15	2.508	497.5	26.44	0.9055	61.16583	104.703
75	4.787	800.6	68.01	0.7983	75.8681	93.27484
120	4.296	1237	68.59	0.8047	84.38157	85.93698
240	4.392	1274	68.21	0.8115	84.83516	75.58961
300	6.019	1411	45.52	0.7552	86.30758	71.93884

Table 2 Electrochemical impedance parameters for ST-37 carbon steel in aerated 1 M HCl solution with $2 \times 10-3$ M of ABT after different immersion times.

Immersion time	$R_s (\Omega \text{ cm}^2)$	$R_{ct} (\Omega \text{ cm}^2)$	$Q_{dl} \times 10^{-6} \; (\mathrm{F \; cm^{-2}})$	n	η%	$C_{dl} \times 10^{-6} \; (\mathrm{F \; cm^{-2}})$
Blank	1.528	193.2	253	0.8143	0	126.9861
15	1.76	359.7	207	0.7917	46.28857	16.82526
75	1.874	421.8	169	0.8163	54.1963	32.59907
120	1.763	583.7	153	0.8079	66.90081	37.69123
240	1.74	728.2	135	0.8005	73.46883	38.67201
300	2.202	767	125	0.8093	74.81095	18.69497

and Noori, 2011). Constant phase element Q_{dl} , R_s and R_{cl} can be corresponded to double layer capacitance, $Q_{dl} = R^{n-1}C_{dl}^n$ solution resistance and charge transfer resistance, respectively.

To corroborate the equivalent circuit, the experimental data are fitted to equivalent circuit and the circuit elements are obtained. Tables 1 and 2 illustrates the equivalent circuit parameters for the impedance spectra of corrosion of steel in solution. From Tables 1 and 2, with increasing the immersion time, increase the solution resistance and charge transfer resistance. Pure HCl has very poor electrical conductivity and is almost an insulator. As can be seen the charge transfer resistance of steel in an HCl solution would be dependent on the solution resistance. As the solution resistance increases with increase in the immersion time (Song and StJohn, 2004). Like

most other organic compounds, MBT and ABT should be easily adsorbed on electrode surface. The double layer capacitance is a good indication of the adsorption of MBT and ABT on steel surface. Q_{dl} can be easily calculated based on the equivalent circuit of the measured EIS. It appears that the capacitance tends to decrease as the immersion time increases. This indicates a change at the steel/solution interface. A decreasing interface capacitance can be caused by high dielectric water at the interface being replaced by some substance that is larger in molecular size. MBT and ABT molecule is larger than water, so the adsorption of the former at the surface of steel can result in a lower Q_{dl} . When the immersion time increases, more inhibitor will be adsorbed on the surface, leading to a lower Q_{dl} . In other words, the steel surface is more

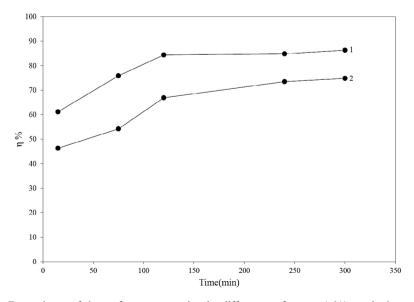


Figure 5 Dependence of the surface coverage by the different surfactants $(\eta\%)$ on the immersion time.

completely covered by inhibitor in a more immersion time, which more effective protects steel from the water corrosion attack. Percent inhibition efficiencies (η) were calculated using the following formula (Eq. (1)).

$$\eta\% = \left[1 - \left(\frac{R_{ct,b}}{R_{ct,i}}\right)\right] \times 100 \tag{2}$$

where $R_{ct,i}$ and $R_{ct,b}$ are values of the charge transfer resistance with and without inhibitor, respectively. As can be seen from Tables 1 and 2 and Fig. 5, the $R_{ct,i}$ values of all the investigated inhibitors increase with increasing time. This is due to increasing surface coverage by increasing inhibitor immersion time. EIS measurements are particularly useful in long time tests because they do not perturb the system dramatically and it is possible to follow the evolution of the inhibitor-metal system over time. The elongations are observed in presence of 2×10^{-3} M inhibitors. The reason of elongation of the semicircles in the Nyquist plots is presence of inhibitor molecules on

the active sites of carbon steel surface. In literature, this kind of Nyquist plots was attributed to all kinds of accumulated species (Dehri and Ozcan, 2006). Explaining scattering is caused by corrosion products of metal and increased with time. The inhibition effectiveness of MBT and ABT was reached maximum at 5th hour, and inhibitor molecules remained on the metal surface even at 5th hours later having the inhibition efficiency was increase. The adsorption of MBT and ABT molecules reduces the anodic dissolution reaction of metal surface areas that exposed to the corrosive media. The accumulation of corrosion products leads to deviation of Nyquist plots from semicircle by increasing the resistance of the diffuse layer (Yurta et al., 2004).

3.2. AFM

Figs. 6–8 depict the morphologies of carbon steel specimens after immersion for 5 h in 1 M HCl solutions without and with

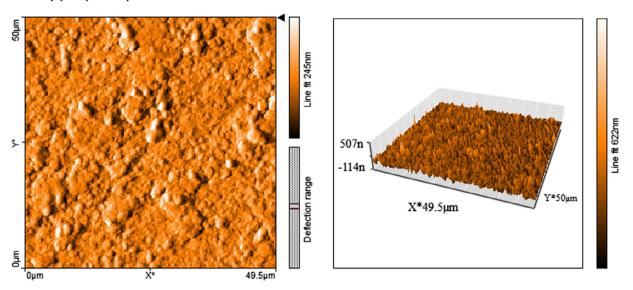


Figure 6 Surface of steel electrode by atomic force microscopy after 5 h immersion at OCP in 1 M HCl.

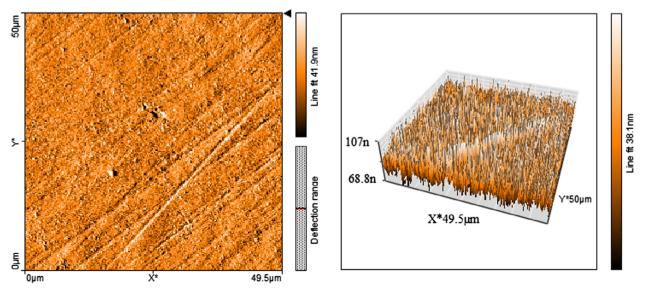


Figure 7 Surface of steel electrode by atomic force microscopy after 5 h immersion at OCP in 1 M HCl and 2×10^{-3} MBT.

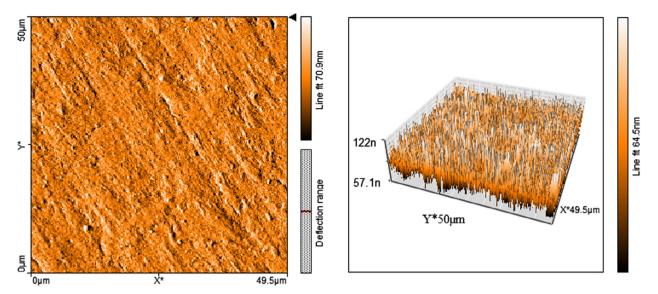


Figure 8 Surface of steel electrode by atomic force microscopy after 5 h immersion at OCP in 1 M HCl and 2×10^{-3} ABT.

 2×10^{-3} M MBT and ABT. In the absence of inhibitor (Fig. 6) the surface displayed a very irregular topography due to corrosion attack. The average roughness R_a of steel in 1 M HCl solution without inhibitor was calculated as 622 nm by atomic force microscopy (Fig. 6). In the presence of MBT and ABT, smoother surface was obtained and the R_a value decreased to 38.1 and 64.5 nm (Figs. 7 and 8) as a consequence of low corrosion damage and the protective formation of an inhibitor layer on steel surface. It can be clearly observed that in the absence of inhibitor, the steel surface was seriously corroded with areas of uniform corrosion (Fig. 6). In the presence of inhibitors, however, the specimen surface was more smooth (Figs. 7 and 8). This is due to the involvement of inhibitor molecules in the interaction with the reaction sites of steel surface, resulting in a decrease in the contact between iron and the aggressive medium and sequentially exhibited excellent inhibition effect.

3.3. The mechanism of inhibition

In order to predict the adsorption mechanism of inhibition onto metal surface, corrosion mechanism of metal must be known. In hydrochloric acid solution the following mechanism proposed for the corrosion of carbon steel (Mulle, 2006).

The anodic dissolution mechanism of metal is:

$$Fe + Cl^- \leftrightarrow (FeCl^-)_{ads}$$
 (3)

$$\left(FeCl^{-}\right)_{ads} \leftrightarrow \left(FeCl\right)_{ads} + e^{-} \tag{4}$$

$$(\text{FeCl})_{\text{ads}} \rightarrow (\text{Fe Cl}^+)_{\text{ads}} + e^-$$
 (5)

$$(\text{Fe Cl}^+)_{\text{ads}} \to \text{Fe}^{2+} + \text{Cl}^- \tag{6}$$

The cathodic hydrogen evolution mechanism is:

$$Fe + H^+ \leftrightarrow (FeH^+)_{ads}$$
 (7)

$$\left(FeH^{+}\right)_{ads}+e^{-}\rightarrow\left(FeH\right)_{ads}\tag{8}$$

$$(FeH)_{ads} + H^+ + e^- \rightarrow Fe + H_2 \tag{9}$$

If one considers the structures of the investigated compounds, several potential sources of inhibitor-metal interaction can be identified. In the case of MBT inhibitor, there

are the free electron pairs on N and O, capable of forming a coordination **σ-bond with Fe (Trabanelli and Carassiti, 1970). Further, the double bonds in the molecule allow back donation of metal d-electrons to the π^* -orbitals, which is a type of interaction that cannot occur, e.g. with amines. And the π -electrons from the aromatic rings may interact with the metal surface. Furthermore, especially in acidic media, electrostatic interaction is possible between the negatively charge of iron surface (which may be brought about by specific adsorption of Cl anions) and the positively charged inhibitor, following protonation of its basic functionalities. In the case of ABT inhibitor, basically the same interactions can occur, with difference that no oxygen atoms. The essential effect of these compounds as corrosion inhibitors is due to the presence of free electron pairs in the N, O and S atoms, π -electrons on the aromatic rings, molecular size, heat of hydrogenation, mode of interaction with the metal surface, and formation of metallic complexes. It is well known that carbon steel has co-ordination affinity toward N, O, and S bearing ligand. Hence, adsorption on carbon steel can be attributed to coordination through hetero-atoms and π -electrons of aromatic rings (Ahmad et al., 2010). In all investigated benzothiazole derivatives, there are unshared electron pairs on N, O, and S, capable of forming **σ-bond with carbon steel. Further, the double bonds in the molecule allow back donation of metal d-electron to the π^* -orbital. Another striking feature for high inhibition performance of all studied compounds is the presence of S-atom. The presence of S-atom in the inhibitor structure makes the formation of $d\pi$ - $d\pi$ bond resulting from the overlapping of 3d-electrons. From carbon steel, the 3d vacant orbital of S-atom become possible, this enhances the adsorption of the compounds on the metal surface. Also the lower solubility of S compounds and the greater polarizability of S atoms increase the inhibition efficiency of these compounds.

The high inhibition efficiency in MBT is due to the presence of other hetero-atom S which has excess free lone pair of electrons able to bond with steel. MBT is known to form a thin polymeric film on steel thus yielding a high inhibitor efficiency

(Ahmad et al., 2010; Patel et al., 2004). In the case of ABT the lone pair of electrons of the "N" atom of the amino group attached to the thiazole ring provides an additional anchoring site for the inhibitor to bind with the metal surface thereby increasing the strength of adsorption and hence the inhibition (Patel et al., 2004).

4. Conclusions

- The addition of MBT and ABT compounds to the 1 M HCl solution reduces corrosion of carbon steel. It was found that MBT is more efficient corrosion inhibitor for carbon steel protection.
- 2. In both the acid media, the inhibitor efficiency of the newly synthesized inhibitors increased with an increase in the immersion time. The high inhibition efficiency of inhibitors was attributed to the formation of a film on the steel surface.
- The maximum inhibitor efficiency for MBT and ABT was observed at 5th hour.
- 4. Impedance measurements indicate that with increasing immersion time, the charge transfer resistance (R_{ct}) increased, while the double-layer capacitance (C_{dt}) decreased.
- 5. The high resolution AFM micrographs showed that the corrosion of carbon steel in 1 M HCl solution was described by corrosion attack and the addition of inhibitor to the aggressive solutions diminished the corrosion of carbon steel.

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